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Electrostatic Double Layer Influence on Gravimetric Separation of Bidisperse Suspensions

by

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fulfillment of the requirements for the degree of Master of Engineering

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Abstract

Gravity separation of bidisperse suspensions consisting of SiO_2 microparticles, nanoparticles, monovalent ions, and water in a square channel was studied experimentally. The microparticles had a density of $2400 \pm 60 \text{ kg/m}^3$ and an average particle size of $98 \text{ }\mu\text{m}$ while the nanoparticles had a density of $2100 \pm 300 \text{ kg/m}^3$ and an average particle size of 12 nm . Ion concentration, in the form of dissolved KCl salt, was varied to manipulate the extent of the electrostatic double layer field surrounding the non-settling nanoparticles. The suspensions were dilute with microparticle and nanoparticle concentrations not exceeding 11 and 0.12 vol. %, respectively. From the experimental data obtained from this study, the size of the electrostatic double layer did not influence the settling velocity of the microparticles.

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I am grateful for having been given the opportunity to work on this project by Dr. J. Masliyah. His technical discussions as well as those with Mr. A. Afacan were invaluable to the completion of this work. Special thanks goes to my parents and Brenda who have all supported me greatly through my university career.

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Introduction

In many industrial applications, processes involving gravity particle separation are widely employed. Such common uses include the purification of municipal drinking water and the concentration of heavy minerals in the mining industry. Since it is desirable to control the settling parameters in a manner that favors the process, it becomes imperative to continually improve upon the understanding of the complex relationships that govern separation. In order to probe the extent of influence that the electrostatic double layer has in gravimetric particle separation, an experiment that involves varying the amount of monovalent ions in a bidisperse mixture of nanoparticles and microparticles in water will be conducted. During the experiment, the time required for the microparticles to settle a predetermined distance will be measured and the resulting settling velocity will be compared with those obtained at different ion concentrations.

Equipment

A clear, graduated glass square channel with dimensions of 20mm x 20mm x 400mm attached to a square base was used to investigate particle settling velocities. The square channel was selected over a cylindrical shape in order to minimize the errors in observations that would be caused by the refraction in curved glass. Furthermore, the width of the square channel was much larger than the diameter of the microparticles that would be placed inside in order to reduce the significance of the wall effect (Fand and Thinakaran, 1990). Since the dimension ratio of the hydraulic diameter of the channel to the diameter of the particles ($D_H / d_L = 204$) was much larger than 100, the wall effect was negligible (Coulson and Richardson, 1978).

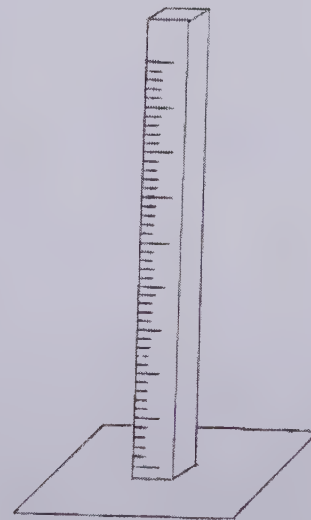


Figure 1. Glass Channel

The spherical silica microparticles were purchased from MANUS Abrasive Systems Inc. (Edmonton, Canada. Stock #071013) and further refined with mechanical sieving. For sieving, three USA standard test sieve trays made by Fisher Scientific with mesh sizes 106, 90 and 75 μm were stacked top to bottom respectively. Approximately 100g of silica particles were loaded each time onto the top tray and the stack was then placed in the Ro-Tap model B testing sieve shaker (TYLER Combustion Engineering Inc.) for a half hour. Subsequently, only the particles accumulated on the 90 μm tray were collected and the sieves were cleaned with compressed air before being reloaded again. Once all the silica had been sifted, the entire process was repeated three more times. The resifting process ensured that the majority of the fine particles less than 90 μm would not be collected for use in the experiment. The final size of the microparticles ($d_{50} = 98 \mu\text{m}$) was measured using a particle size analyzer (Mastersizer 2000) manufactured by Malvern Instruments and shown to have the size distribution as illustrated in Figure 2. The density was determined by measuring the volume of displaced water from a known mass and found to be $2400 \pm 60 \text{kg/m}^3$, which agreed with the published range of $2200 - 2650 \text{kg/m}^3$ (Perry, 1997).

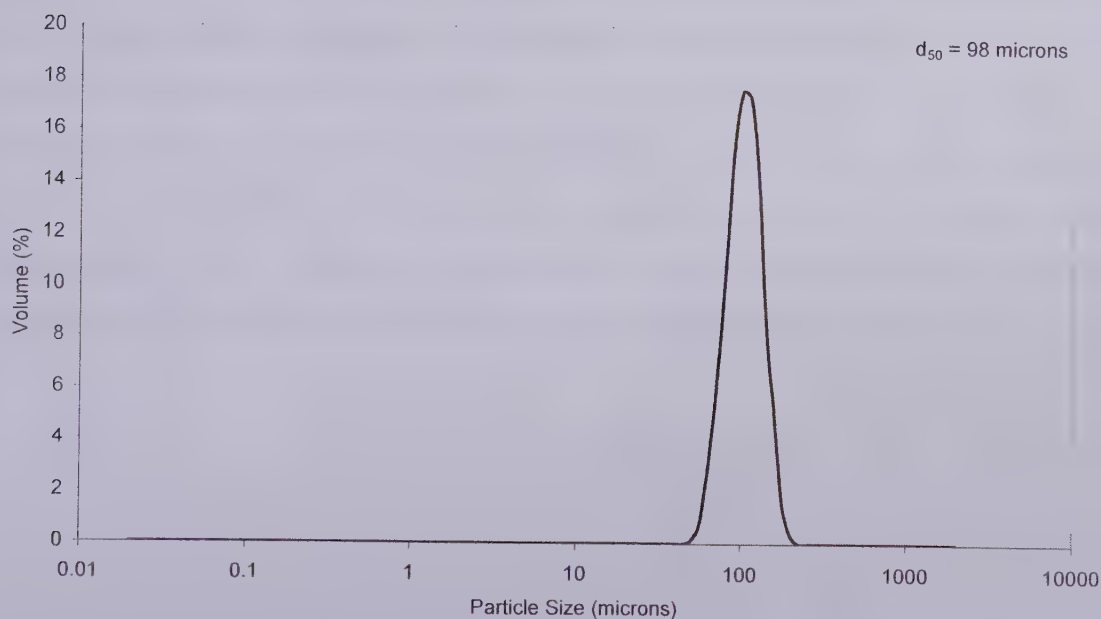


Figure 2. Particle Size Distribution of Microparticles

The nanoparticles were manufactured by Degussa under the product name Aerosil 200. They were hydrophilic fumed silica particles with an average particle size of 12 nm. The density was determined by creating a dilute suspension using a known mass of nanoparticles and water and then measuring the density of the suspension with a density meter. Equation 1 was then used to calculate the nanoparticle density, which was found to be $2100 \pm 300 \text{ kg/m}^3$, which agreed well with the reported literature value of 2200 kg/m^3 (Holland, 2005). The large relative error in the determined density was due to the limited precision in measuring the density of mixtures with low solids fractions.

$$\rho_s = \frac{x_s}{\left(\frac{1}{\rho_M} - \frac{1-x_s}{\rho_f} \right)} \quad (1)$$

Deionized water used throughout the experiment was obtained using the Ultra Pure Water System manufactured by Millipore Corporation (Milli-Q UV Plus with QPAK1). Ion concentrations in the mixtures were provided by the dissolution of (certified A.C.S.) potassium chloride salt ordered directly from Fisher Chemicals.

The mass of salt and silica particles were measured using a Sartorius CP3245 electronic balance having accuracy of 0.0001g. However, because of the maximum limit of the balance (200g), a Mettler Toledo PB3002-S electronic balance ($\pm 0.01\text{g}$) was used to measure the mass of water. Fluid densities were measured using an Anton Paar DMA 38 density meter set at 21.0 °C. Additional fluid properties such as surface tension and viscosity were evaluated in a temperature controlled room at 22 °C using a KRÜSS manufactured K100 Processor Tensiometer and a Cannon-Fenske viscometer, respectively. The following calibration equation was used for the viscometer:

$$\mu = t \times 4.150 \times 10^{-9} \times \rho_f \quad (2)$$

Procedure

The Sartorius balance was used to weigh the proper amounts of microparticles and nanoparticles as per concentrations shown in Table 1 based on a total volume of 150mL. The weighed particles were placed in the square channel and using the Mettler balance, the remaining mass consisting of deionized water was added. All the weight measurements were recorded and the top of the square channel was enclosed using Parafilm to prevent evaporation. The prepared mixture was left for 1h to allow the particles to be evenly wetted by the water and for temperatures to become uniform, thus avoiding convection currents (Richardson and Meikle, 1961). The mixture in the column was then manually agitated for approximately 80s with continual inversions at a rate of about 0.5Hz to ensure a uniform distribution throughout. During mixing, it was important to avoid prolonged direct contact with any one area of the glass as it would cause localized heating. After the last inversion, the column was set in the upright position and a settling interface between the microparticle-containing and non-microparticle-containing regions was observed. Since the particles were sifted to fall within a narrow size range, the interface was sharp and delineated. A stopwatch was started when the interface reached the 320mm marking etched on the column and stopped when it reached the 170mm marking. Although the total height was 400mm, only the 150mm region (between the 320mm and 170mm etched markings) was used to collect settling velocity data. The zone above this region allowed time for the unsteady motions of the particles induced by mixing to dissipate, and the zone below included the sludge zone and was avoided due to its different settling behavior (Letterman et al., 1999).

Table 1. Volume Percent of Particles in Investigated Mixtures

	Mixture 1	Mixture 2	Mixture 3	Mixture 4	Mixture 5	Mixture 6
Micro-particles Vol. %	11	11	10	6	6	6
Nano-particles Vol. %	0.12	0.12	0.05	0.12	0.12	0.05

In an effort to minimize errors, the mixing and timing procedures were repeated 15 times for each mixture and an average was obtained. It was found that the recorded times were quite reproducible as they followed a normal distribution with an average standard deviation of 0.32s. After the 15 runs, the amount of pure salt needed to produce a 1×10^{-4} M mixture of KCl was measured on the Sartorius balance and added into the column. It was assumed that the Vol. % of the particles was negligibly affected by the addition of the salt and that only the monovalent ion molarity was increased. Following the same mixing and timing procedures as before, another 15 runs were performed with the particles and salt mixture. Once completed, the salt concentrations were increased twice more (1×10^{-3} M and 1×10^{-2} M of KCl) and 15 timing runs were performed at each of the increased salt concentrations.

In the aforementioned procedure, several assumptions were made that required verification. Firstly, the settling velocity that was determined from the time taken for the interface to travel the full 150mm was the average velocity. The use of the average velocity implied that the velocity was constant through the 150mm distance. Shown in Figure 3, it was experimentally confirmed that the velocity was constant by measuring the time it took for the interface to travel 10mm increments of the 150mm. A constant velocity means that the use of the average velocity was valid and also confirms that the sludge zone had been avoided.

Secondly, the settling velocity of the nanoparticles relative to that of the microparticles was never measured. It was assumed that the colloidal suspension of nanoparticles in water was non-settling. To verify the assumption, the settling velocities of nanoparticle suspensions in water without microparticles present in the square channel were measured, and the results are shown in Table 2. It was determined that the velocities were much slower compared to those of the microparticles (in the order of 1000 times) and thus it could be assumed that the colloidal suspension was non-settling in the context of the experiment.

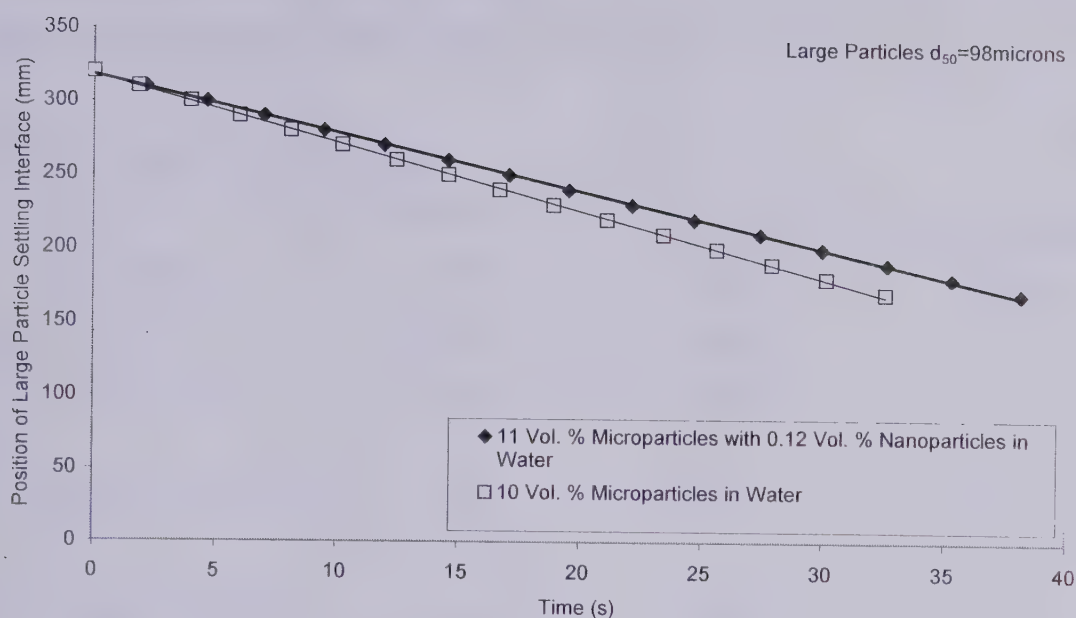


Figure 3. Position of Large Particle Settling Interface as a Function of Time

Table 2. Nanoparticle Settling Velocities in Monodisperse Systems

Mixture	Settling Velocity of Nanoparticles
	(mm/s)
0.12 Vol. % nanoparticles	0.00252
0.12 Vol. % nanoparticles with 0.01M KCl	0.00202
0.05 Vol. % nanoparticles	0.00410
0.05 Vol. % nanoparticles with 0.01M KCl	0.00433

Lastly, to verify that the addition of KCl salt to water containing nanoparticles had the intended effect of only modifying the extent of the electrostatic double layer surrounding the nanoparticles (which would be evident by changes in measured fluid viscosity), several fluid properties such as viscosity, density and surface tension were measured. The properties are summarized in Table 3. The measured values showed that pure water had the lowest viscosity and water with nanoparticles and no KCl had the highest. As expected, water with nanoparticles and KCl had a viscosity in between due to compression of the electrostatic double layer, which will be later discussed in further detail. The fluid densities were inevitably changed with the addition of nanoparticles and

KCl salt. However, the percentage of change was small (0.16 %) and should be minor in its affects on the results of the experiment. Surface tension measurements showed that there was little change due to the addition of KCl salt.

Table 3. Measured Fluid Properties

Fluid	Viscosity @ 22°C (mPa · s)	Density @ 21.0°C (kg/m ³)	Surface Tension (mN/m)
Pure Water	0.961	998.0	72.34
Water and 0.01M KCl	0.961	998.2	72.36
Water and 0.12 Vol.% 12nm particles	1.00	999.4	NA
Water, 0.12 Vol.% 12nm particles and 0.01M KCl	0.962	999.6	NA

In order to gauge the reliability of the results obtained from the experiment, the settling velocities that were determined were compared to those predicted by theory. The results of the comparison are later shown and discussed. The following equation, given by Richardson and Coulson (1978), was used to predict the settling velocity of the microparticles:

$$u_{ML} = e^{n_L-1} [u_{OL}(1 - C_L) - u_{OS}C_S] \quad (3)$$

Where

$$e = 1 - (C_L + C_S) \quad (4)$$

For $0.2 < Re < 1$ (Microparticle $Re = 0.646$)

(From Richardson and Zaki, 1954)

$$n_L = (4.36 + 17.6 \frac{d_L}{D_H}) Re^{-0.03} \quad (5)$$

$$Re = \frac{\rho_f u_{oi} d_i}{\mu_f} \quad (6)$$

$$u_{oi} = \frac{Re_o' \mu_f}{\rho_f d_i} \quad (7)$$

For $Ga < 3.6$ (Nanoparticle $Ga = 1.93 \times 10^{-11}$)

$$Ga = 18 Re_o' \quad (8)$$

For $3.6 < Ga < 10^5$ (Microparticle $Ga = 12.9$)

$$Ga = 18 Re_o' + 2.7 Re_o'^{1.687} \quad (9)$$

$$Ga = \frac{d_i^3 \rho_f g (\rho_s - \rho_f)}{\mu_f^2} \quad (10)$$

An alternate equation proposed by Patwardhan and Tien (1985) was given as:

$$u_{ML} = \frac{u_{OL} e^{n_L - 2} (1 - C_L) (\rho_L - \rho_M)}{(\rho_L - \rho_f)} - \frac{u_{OS} e^{n_S - 2} C_S (\rho_S - \rho_M)}{(\rho_S - \rho_f)} \quad (11)$$

Where

For $Re < 0.2$ (Nanoparticle $Re = 1.07 \times 10^{-12}$)

(From Richardson and Zaki, 1954)

$$n_s = 4.65 + 19.5 \frac{d_s}{D_H} \quad (12)$$

$$\rho_M = \rho_f(1 - C_L - C_S) + \rho_S C_S + \rho_L C_L \quad (13)$$

A modified version of Equation 3, proposed by Mirza and Richardson (1979) was as follows:

$$u_{ML} = u_{OL} * e^{n_L + 0.4 - 1} (1 - C_L) - u_{OS} * e^{n_S + 0.4 - 1} C_S \quad (14)$$

Results and Discussion

Data from this experiment can be found in Appendix A.

Figures 4 and 5 show the settling velocity of microparticles in a mixture at various ion and nanoparticle concentrations. The concentrations of the ions were assumed to be directly proportional to the amount of salt present, as dissociation of KCl into monovalent ions occurs in water (@ 20°C until 4.5 M (Perry, 1997)). Both figures show that the velocities, for the particles and volume fractions investigated, were not affected by changes in salt concentrations. This was an unexpected trend, as increased ions result in the compression of the electrostatic double layer surrounding the nanoparticles in suspension. A smaller electrostatic double layer would cause the shear viscosity of the mixture to decrease, which should lead to faster settling times. The hypothesis was that the effects of the slight viscosity changes were overshadowed by the dominating gravity, drag and buoyancy forces acting upon the microparticles. Also shown in both figures, varied nanoparticle concentrations did not significantly affect settling rates. The rates were not affected since there was no appreciable change in the amount of water that flowed upwards. The flow of water, normally induced by displacement caused by settling particles, remained constant because the nanoparticles were non-settling.

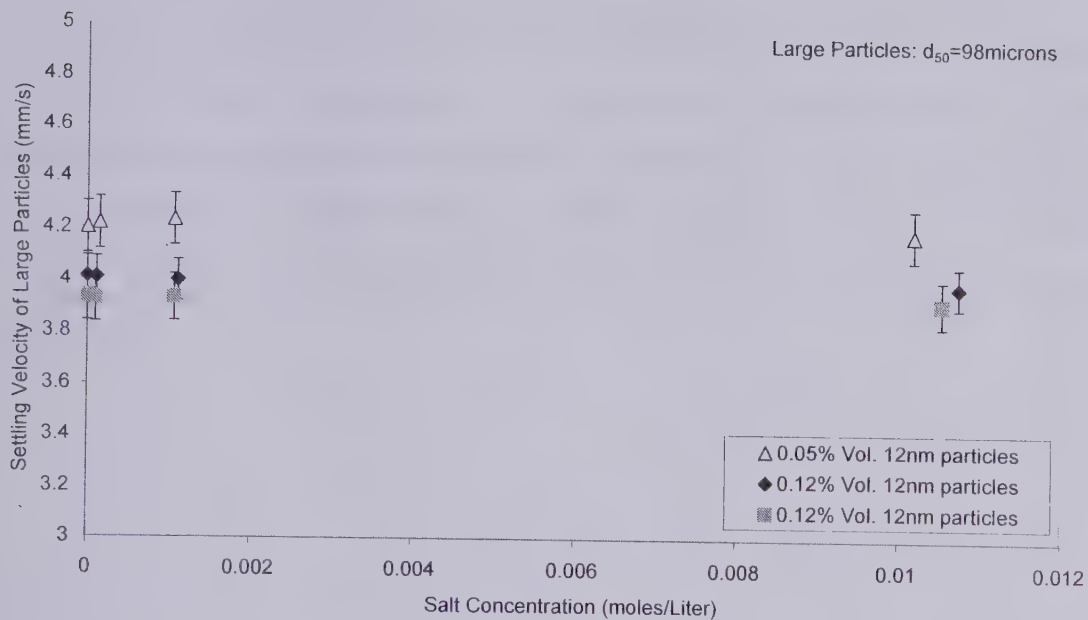


Figure 4. Effect of Small Particle Concentration with 11% Vol. Large Particles

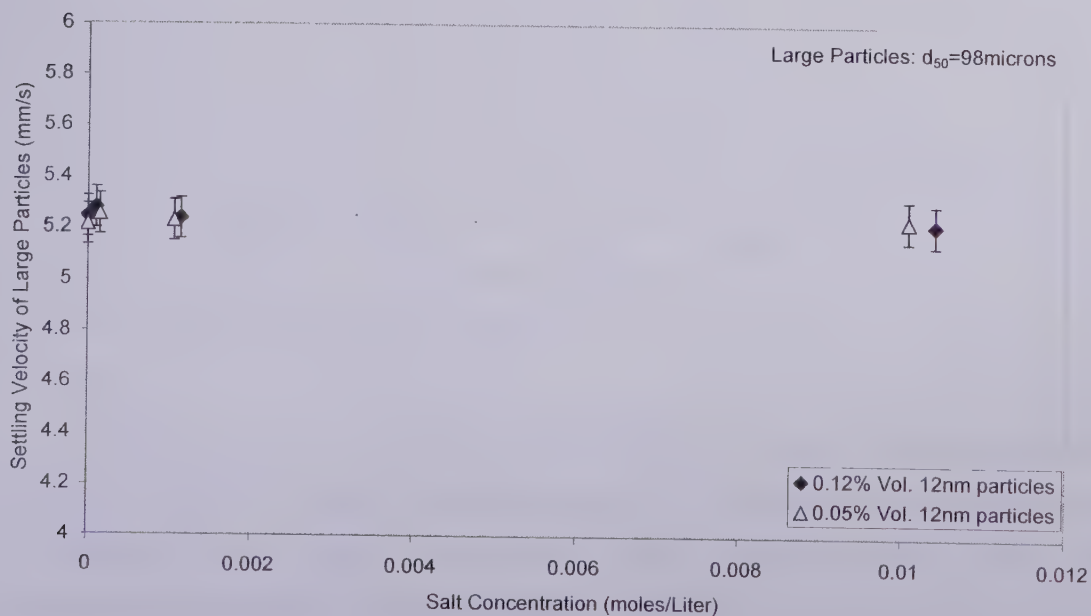


Figure 5. Effect of Small Particle Concentration with 6% Vol. Large Particles

Figures 6 and 7 show the settling velocity as a function of microparticle and ion concentrations while nanoparticle concentrations were kept constant. Similarly, the measured values demonstrate that the velocities were not affected by changes in ion concentration. However, it was evident that modifying microparticle concentrations significantly affected settling rates. This was an expected trend as increased microparticle amounts caused larger volumes of fluid to be displaced during settling and also decreased the free area between neighboring particles. This led to an increased upwards velocity gradient in the fluid close to the particles that subsequently slowed the settling rates.

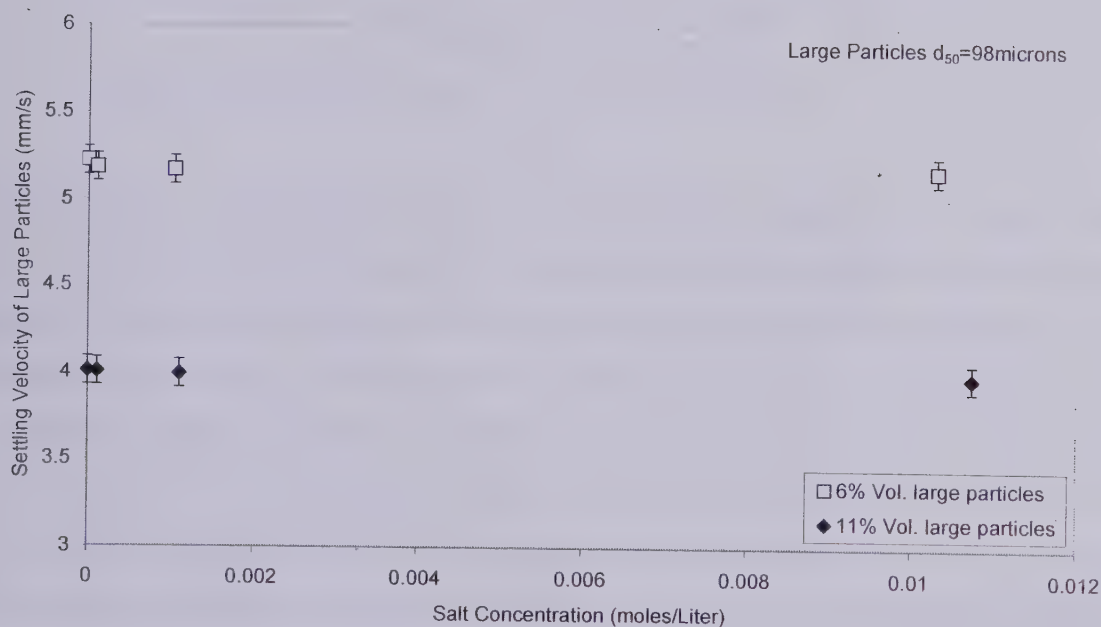


Figure 6. Effect of Large Particle Concentration with 0.12% Vol. 12nm Particles

Figure 8 shows a comparison of the settling velocity of the microparticles at varying concentrations within a bidisperse mixture. As consistent with Figures 6 and 7, the measured values show an increasing settling velocity trend as microparticle concentrations decreased. The experimental data are compared to those predicted by theory to determine if the values obtained lie within the expected range given in literature. The values and trend agree well with those predicted by Patwardhan and Tien (1985), and Coulson and Richardson (1978) with maximum errors of 6.2% and 6.5%.

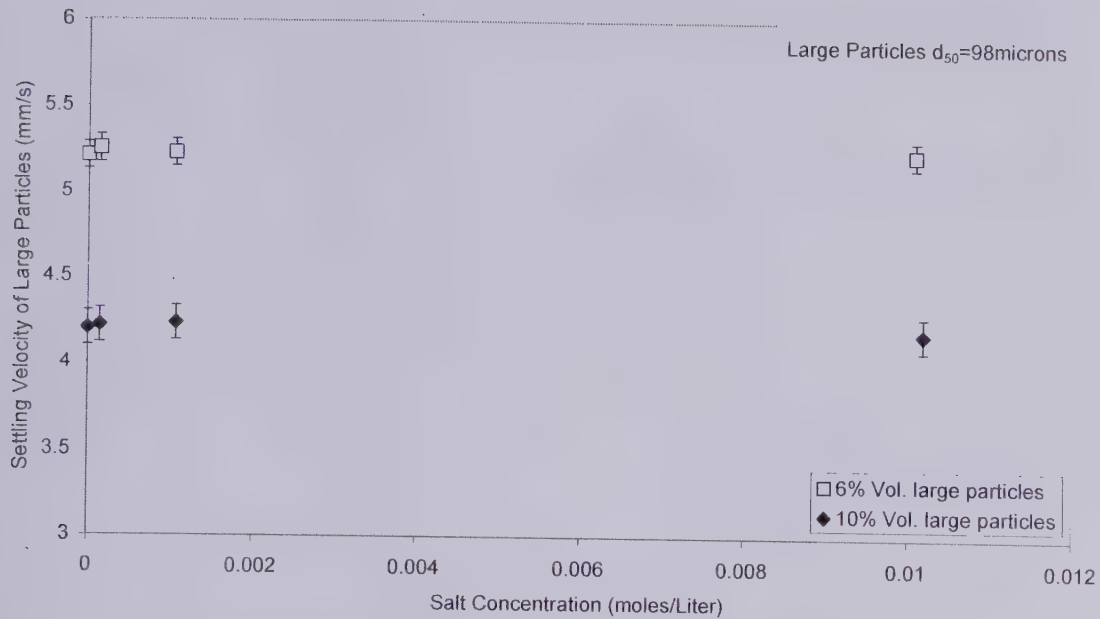


Figure 7. Effect of Large Particle Concentration with 0.05% Vol. 12nm Particles

respectively. The strong agreement with the two theories validates that the data obtained from this experiment were credible. However, using the empirically modified formula given by Mirza and Richardson (1979), the predicted values underestimated the measured settling velocities with a maximum error of 9.4%. In conflict from the findings of this experiment, studies from Mirza and Richardson and Selim et al. (1983) found that the equations given by Coulson and Richardson (adapted from Richardson and Zaki, 1954) overpredicted settling rates by 5 – 50% because the particle interactions that would act to slow the larger particles were not accounted for. Thus, an experiment (using particles of the same density and sizes of equal magnitude) was conducted by Mirza and Richardson to determine the correction factor of $(\text{voidage})^{0.4}$. In the case of this experiment, the factor served to overcorrect the predicted values and led to predictions lower than the observed velocities. This was caused by the fact that in the current experiment, while the densities of the microparticles and nanoparticles were similar, there was such a large difference between their sizes (in the order of 10,000 times) that the interparticle collisions between the large and small particles would negligibly affect the settling velocity of the larger particles.

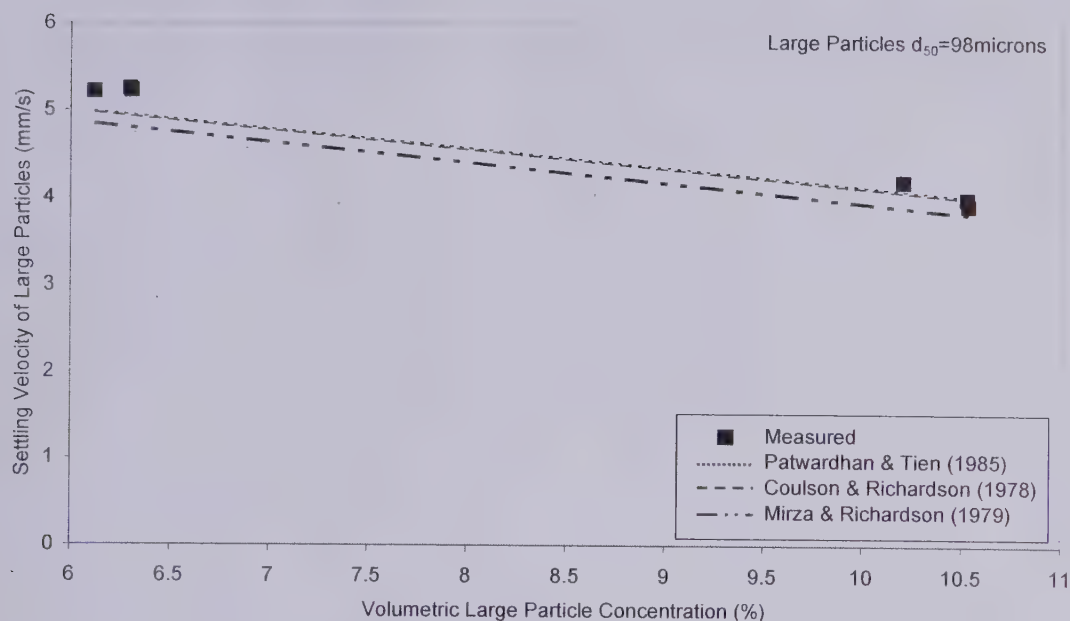


Figure 8. Settling Velocity of Microparticles in a Bidisperse Mixture Without Addition of Ions

Conclusions

The settling velocities of microparticles in dilute bidisperse mixtures with nanoparticles were studied. The microparticles and nanoparticles were SiO_2 with average particle sizes of $98\ \mu\text{m}$ and $12\ \text{nm}$, respectively. Ion concentration (in the form of dissolved KCl salt) was varied for each mixture and each mixture was composed of different concentrations of microparticles and nanoparticles not exceeding 11 and 0.12 vol. %, respectively. The following conclusions were found:

- Varying the salt concentrations between 0 and 0.01M did not affect settling velocities. Higher salt concentrations were known to compress the electrostatic double layer surrounding the nanoparticles. Thus, the resultant changes in the extent of the double layer did not affect settling velocities.
- Settling velocities were not significantly affected by changes in 12nm particle concentrations in the dilute range investigated (0.05 – 0.12 Vol. %).
- Increasing large particle concentrations (from 6 – 11 Vol. %) significantly decreased settling velocities.

- The velocities predicted by Patwardhan and Tien, and Coulson and Richardson equations matched well with the experimental results. However, Mirza and Richardson underpredicted the velocities due to an overcorrection for particle collisions.

Nomenclature

C	Volume fraction
d	Diameter of the particle, m
D _H	Hydraulic diameter of the glass channel, m
e	Volume void fraction
g	Gravity, 9.81 m/s ²
Ga	Galileo number
n	Richardson and Zaki exponent
Re	Reynold's number
Re'	Modified Reynold's number
t	Time, s
u	Velocity, m/s
x	Mass fraction

Greek Symbols

μ	Dynamic viscosity, Pa's
ρ	Density, kg/m ³

Subscripts

f	Fluid
i	ith component in the mixture
L	Microparticle
M	Mixture
O	Terminal (velocity)
s	Nanoparticle

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Appendix A

Mixture 1 with 0.12 Vol. % Nanoparticles and 11 Vol. % Microparticles

Run #	Recorded Time (s)			
	0M Salt	1×10^{-4} Salt	1×10^{-3} Salt	1×10^{-2} Salt
1	37.46	38.91	38.4	38.89
2	38.43	38.35	38.18	38.73
3	37.56	37.98	38.5	38.79
4	38.5	38.68	37.64	38.52
5	37.82	38.77	37.83	38.11
6	38.17	38.35	38.38	37.21
7	38.18	37.32	38.91	38.12
8	38.69	38.5	37.7	38.58
9	38.43	38.58	38.6	38.41
10	38.24	38.02	37.81	38.77
11	38.85	37.88	38.12	38.62
12	38.28	38.34	37.62	38.54
13	37.66	38.24	38.55	38.02
14	38.21	37.41	37.77	37.28
15	37.95	37.76	38.51	37.83

Mixture 2 with 0.12 Vol. % Nanoparticles and 11 Vol. % Microparticles

Run #	Recorded Time (s)			
	0M Salt	1×10^{-4} Salt	1×10^{-3} Salt	1×10^{-2} Salt
1	37.62	37.48	36.9	37.9
2	37.66	37.28	38.45	38.31
3	37.36	37.62	37.39	37.72
4	36.94	37.49	37.29	37.18
5	37.14	37.88	37.54	38.05
6	36.96	37.75	37.48	37.39

7	37.09	37.86	37.44	38.28
8	37.52	37.41	37.91	37.46
9	36.85	36.6	37.55	37.22
10	37.79	37.13	37.19	38.11
11	37.8	36.95	37.87	37.32
12	37.58	37.6	37.68	37.29
13	37.69	37.21	36.78	37.87
14	37.82	37.27	37.71	37.08
15	37.12	37.89	37.74	37.95

Mixture 3 with 0.05 Vol. % Nanoparticles and 10 Vol. % Microparticles

Run #	Recorded Time (s)			
	0M Salt	1×10^{-4} Salt	1×10^{-3} Salt	1×10^{-2} Salt
1	35.3	35.95	34.77	36.29
2	36.46	35.66	35.16	36.1
3	35.7	35.84	34.69	35.74
4	35.19	35.43	35.96	36.17
5	35.66	35.92	35.69	36.65
6	36.52	35.28	35.33	36.33
7	35.46	34.86	35.62	36.32
8	35.64	35.86	35.48	35.62
9	36.22	35.93	35.23	35.48
10	35.37	34.76	36.31	35.27
11	35.95	35.39	35.46	35.42
12	35.43	35.56	35.06	35.56
13	36.2	35.67	35.16	35.79
14	35.24	35.44	35.84	35.29
15	35.16	35.92	35.77	35.83

Mixture 4 with 0.12 Vol. % Nanoparticles and 6 Vol. % Microparticles

Run #	Recorded Time (s)			
	0M Salt	1×10^{-4} Salt	1×10^{-3} Salt	1×10^{-2} Salt
1	28.54	28.37	28.69	28.58
2	28.69	28.38	28.2	28.7
3	29.08	28.45	28.42	28.66
4	28.65	28.76	29.02	28.72
5	28.48	28.39	28.98	28.96
6	28.41	28.46	28.63	28.94
7	29.02	28.47	28.56	28.36
8	28.44	28.57	28.46	28.98
9	28.5	28.3	28.76	28.45
10	28.35	28.5	28.64	28.56
11	28.31	28.32	28.31	29.02
12	28.4	28.36	28.87	29.11
13	28.86	28.52	28.79	28.5
14	28.73	28.36	28.56	28.68
15	28.62	28.16	28.74	28.96

Mixture 5 with 0.12 Vol. % Nanoparticles and 6 Vol. % Microparticles

Run #	Recorded Time (s)			
	0M Salt	1×10^{-4} Salt	1×10^{-3} Salt	1×10^{-2} Salt
1	28.45	28.62	29.16	28.98
2	28.72	29.98	29.25	29.18
3	28.56	29.16	28.92	28.83
4	28.48	29.1	29.23	28.84
5	28.81	29.06	29.16	28.71
6	28.91	28.63	29.12	28.85
7	28.87	28.84	28.91	29.21
8	28.8	29.11	28.81	29.24

9	28.49	28.51	28.91	28.84
10	28.96	29.16	29.22	28.9
11	29.02	28.97	28.94	28.96
12	29.04	28.56	28.87	28.94
13	28.52	28.72	29.1	29.12
14	28.77	28.92	28.76	29.03
15	28.5	28.7	28.82	29.12

Mixture 6 with 0.05 Vol. % Nanoparticles and 6 Vol. % Microparticles

Run #	Recorded Time (s)			
	0M Salt	1×10^{-4} Salt	1×10^{-3} Salt	1×10^{-2} Salt
1	28.58	28.38	28.48	28.8
2	29.06	28.56	28.93	29.11
3	28.96	28.61	28.67	29.13
4	28.72	28.45	28.76	28.78
5	29.05	28.44	29.01	28.52
6	28.62	28.32	28.87	28.18
7	29.03	28.67	28.41	28.61
8	28.63	28.65	28.49	28.57
9	29.11	28.77	28.72	28.83
10	29.16	28.54	28.43	28.45
11	28.54	28.72	28.36	28.32
12	28.39	28.34	28.83	28.77
13	28.51	28.79	28.78	28.39
14	28.86	28.37	28.94	28.8
15	28.47	28.71	28.6	28.61

